

## EXAMPLE 11

Preparation of a dentin-adhesive bicomponent mixture

Equal parts of an activator resin consisting of  
55 parts bis-GMA  
45 parts TEDMA  
3 parts N,N-bis-hydroxyethyl-p-toluidine  
and of a catalyst resin consisting of  
55 parts bis-GMA  
45 parts TEDMA  
3 parts benzoyl peroxide  
5 parts polymethacrylated polyboric acid (Ex. 10)  
were mixed together. The mixture (I) was applied thin to dentin and cured along with a cylinder of Composite Merz.

A comparison was carried out with a mixture (II) of activator and catalyst resins but without the polymethacrylated polyboric acid.

Subsequent to 24 hours in water there was an extensive difference in adhesion to the dentin, with the samples of mixture I exhibiting a mean tensile strength of 7.82 N/mm<sup>2</sup> and those of mixture II one of only 0.54 N/mm<sup>2</sup>.

## EXAMPLE 12

Preparation of a polymethacrylated polysulfonate

5.4 g of hydroxyethyl methacrylate, 10.1 g of potassium methacryloylpropylsulfate, and 1.6 g of lauroyl peroxide were heated in 80 ml of methyl alcohol and 20 ml of toluene at 65° C. until termination of precipitation. The batch was filtered and the filtrate washed with hexane and dried.

The yield was 6.4 g of a water-soluble copolymer with a molecular weight of 7490, corresponding to approximately 20 units for each of the monomers employed.

1.88 g of the copolymer was stirred along with 0.54 g of methacrylic-acid chloride and 0.50 g of triethylamine in 50 ml of tetrahydrofuran for 4 days at room temperature. The precipitate was washed with hexane and dried.

The yield was 1.92 g of a white, water-soluble, powdery polymethacrylic polypotassium sulfonate, continuing to exhibit a C=C band at 1640 cm<sup>-1</sup>. The potassium content was 7.9% and the sulfur content 6.2%.

## EXAMPLE 13

Preparation of a light-curing adhesive mixture

A mixture of  
55 parts bis-GMA  
45 parts TEDMA  
1 part butyl dimethylaniline  
1 part camphor quinone  
5 parts polymethacrylic polypotassium sulfonate (Ex. 12)

is tested on bovine dentin as described with reference to Example 9. The result was a mean bond strength of 5.8 N/mm<sup>2</sup>.

## EXAMPLE 14

Agents for bonding methacrylate-based composites to oxidic, mineral, ceramic, vitreous, and metallic substrates

A light-curing mixture of  
50 parts bis-GMA  
50 parts TEDMA

10 parts polymethacrylated oligomaleic acid (Ex. 4)  
1 part butyl dimethylaniline  
1 part camphor quinone

was applied thin to a polished surface of the following substrates and irradiated under a halide light (450-500 nm) for 2 minutes along with cylinders (4 mm in diameter and 6 mm high) of Composite Merz, Light-curing.

After 24 hours in water at 37° C., tensile-strength tests indicated the following bond strength. Control results from tests of the same mixture without the components in accordance with the invention are indicated in parentheses.

	N/mm <sup>2</sup>	
Ionomer cement	5.2	(1.2)
Silicate cement	17.8	(0.9)
Glass	12.1	(0.3)
Porcelain	19.9	(0.1)
Cobalt-chromium alloy	9.2	(0.9)
Gold-platinum alloy	3.6	(0.2)

## EXAMPLE 15

Preparation of a prepolymeric polymethacrylated polymaleic acid

60 g of maleic acid anhydride and 9 g of lauroyl peroxide were refluxed for 4 days in 150 ml of tetrahydrofuran. The tetrahydrofuran was extracted and the resulting viscous oil washed with hexane.

The polymaleic acid anhydride has a molecular weight of 1850, corresponding to approximately 17 units. The IR spectrum was identical with that of the oligomaleic acid anhydride from Example 4.

9.8 g of the oil were dissolved in 30 ml of THF and stirred with 12 g of hydroxyethyl methacrylate for two weeks. The THF was extracted, leaving a viscous oil of polymethacrylated polymaleic acid with an IR spectrum identical to that of the polymethacrylated oligomaleic acid from Example 4.

## EXAMPLE 16

A bonding agent

A mixture of  
50 parts bis-GMA  
50 parts TEDMA  
10 parts polymethacrylated polymaleic acid (Ex. 15)  
1 part butyl dimethylaniline  
1 part camphor quinone

is tested by the same procedure described with reference to Example 9. The mean bond strength is 6.8 N/mm<sup>2</sup>.

## EXAMPLE 17

Preparation of a prepolymeric polymethacrylated polyaldehydopolymaleic acid

9.8 g of polymaleic acid anhydride are dissolved in 30 ml of THF, treated with 6 g of hydroxyethyl methacrylate, and stirred for 2 weeks under an HCl atmosphere until free hydroxyethyl methacrylate can no longer be detected. 7.5 g of aminobutyric-aldehyde diethylacetal and a little triethylamine are added and the batch stirred for 3 more days.

The white precipitate was filtered out, the THF extracted, and the remaining oil washed with hexane, dilute HCl, and water, and dried. The oil (polymethacrylic-polyaldehydopolymaleic acid) reduces an alka-